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EVALUATION OF INDENTATION HARDNESS PROPERTIES AND INDENTATION METHODOLOGY FOR DETERMINING THE INFLUENCE OF CHEMICAL EXPOSURE ON POLYMERIC MATERIALS

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13. ABSTRACT *(Maximum 200 words)*
The relevance and importance of the indentation experiment in chemical-material compatibility testing was evaluated. The spectrum of methods and instrumentation was surveyed. The ranking of methods was thermomechanical analysis (TMA), International Rubber Hardness Degree (IRHD), Shore A/D ISO Ball, and Rockwell. Based on the advantages of the TMA method, a further review of indentation instrumentation is provided.

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PREFACE

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EVALUATION OF INDENTATION HARDNESS PROPERTIES AND INDENTATION METHODOLOGY FOR DETERMINING THE INFLUENCE OF CHEMICAL EXPOSURE ON POLYMERIC MATERIALS

1. INTRODUCTION

The determination of chemical effects on materials is of primary importance in the development of modern military systems that are survivable in a chemically contaminated battlefield environment. The AR 70-71 and DoD Directive 5000 specifically address the nuclear, biological, and chemical (NBC) requirements in decontaminability, hardness, and compatibility. This report specifically applies to methods appropriate to the Nuclear, Biological, and Chemical Contamination Survivability (NBCCS) Hardness criterion.

Note that a nomenclature difficulty arises from the dual use of the term "hardness." Therefore, to specify the NBCCS Hardness criterion, we will employ a capitalized "Hardness." For the hardness test method based on indentation, we will substitute "indentation test" and/or employ the uncapitalized "hardness" term.

2. REVIEW

Approximately 20-30 variations on hardness tests were surveyed by performing key word literature searches on various hardness methods and terms. Appropriate journal articles, instrumentation literature, and standard American Society for Testing and Materials (ASTM) and International Standardization Organization (ISO) test methods were obtained and reviewed. The specific areas that follow were reviewed.

- Current status of hardness property testing and instrumentation
- Trends toward less arbitrary test scales
- Use of hardness testing in chemical compatibility studies
- Correlation of hardness tests with other mechanical tests
- Correlation of the effects of liquids on hardness versus other mechanical properties

3. EVALUATION RESULTS

3.1 Candidate Indentation Tests and Scales for Chemical Effect Studies.

The major hardness scales identified were Shore, International Rubber Hardness Degrees (IRHD), Ball Indentation, Rockwell, Barcol, Vickers, Brinell, and Knoop for materials ranging from very soft (foam) to rigid plastics. The ASTM and/or ISO standard methods were identified for the Shore, IRHD, Ball Indentation, Rockwell, and Barcol scales. Subsequently, the Vickers, Brinell, and Knoop scales were ranked lower in the evaluation. The Survey results of general hardness scales, specimen physical states, and standard method numbers for scales evaluated are listed in Table 1. The hardness scale is listed in column 1, the specimen physical state in column 2, and applicable standard test numbers in column 3. There was more specific interest in the hardness scales for which there was an ASTM and an ISO standard test method (Shore, IRHD, and Rockwell).

Table 1. Survey of Hardness Scales, Specimen Physical States, and Test Numbers

Hardness Scale	Specimen Physical State	Test Numbers
Shore	Foam to rigid plastics	ASTM D2240, ISO 868
IRHD	Soft to hard rubbers	ASTM D1415, ISO's 1818, 48, 1400
Ball Indentation	Plastics	ISO 2039-1
Rockwell	Plastics	ASTM D785, ISO 2039-2
Barcol	Rigid Plastics	ASTM D2583
Vickers	Rubber to plastics	-
Brinell	Rubber to plastics	-
Knoop	Plastics	-

3.2 Thermomechanical Analysis Overview.

The thermomechanical analysis (TMA) is included in this evaluation, because TMA can provide temperature dependent data for the Department of Department -40 to +160 °F criteria and TMA uses small specimens and, therefore, smaller contaminant volumes.

A brief description of a typical TMA is shown in Figures 1-5. Figure 1 is a diagram of the front of the main TMA module, showing the key operational features such as the probe jacket, furnace, and weight platform (under the weight platform cover). Figure 2 shows the probe assembly in more detail, such as the location of the thermocouple. Figure 3 displays the bottom of the probe assembly, showing the relative position of the sample, probe, and thermocouple. Figure 4 shows the probe options typically available for the TMA. Finally, Figure 5 shows the position of the probe assembly within the TMA furnace assembly.

Text continues on page 14.

Item	Description
1	Rigid back plates
2	Base
3	Perspex lid for balance cover (hinged for easy access)
4	Gas-tight balance cover under perspex lid
5	Weight platform cover
6	Mounting plate for balance
7	'dl/dt output' control knob
8	'air/gas flow' control knob
9	'power' button (main button)
10	'reset' button for audible alarm system
11	'hold' button for motorized furnace mechanism
12	'furnace' button (up/down button) for motorized furnace mechanism
13	Screw height adjuster
14	Graduated flow-meter for gas
15	Quartz water-cooled jacket of probe or fiber attachment assembly
16	Water-cooled furnace
17	Removable furnace cover
18	Location of level bubble indicator
19	Front foot (fixed)
20	Thumbwheels for adjustable rear feet
21	Microswitch connection for motorized furnace mechanism
22	Locking screws
23	Metal collar
24	Transducer housing

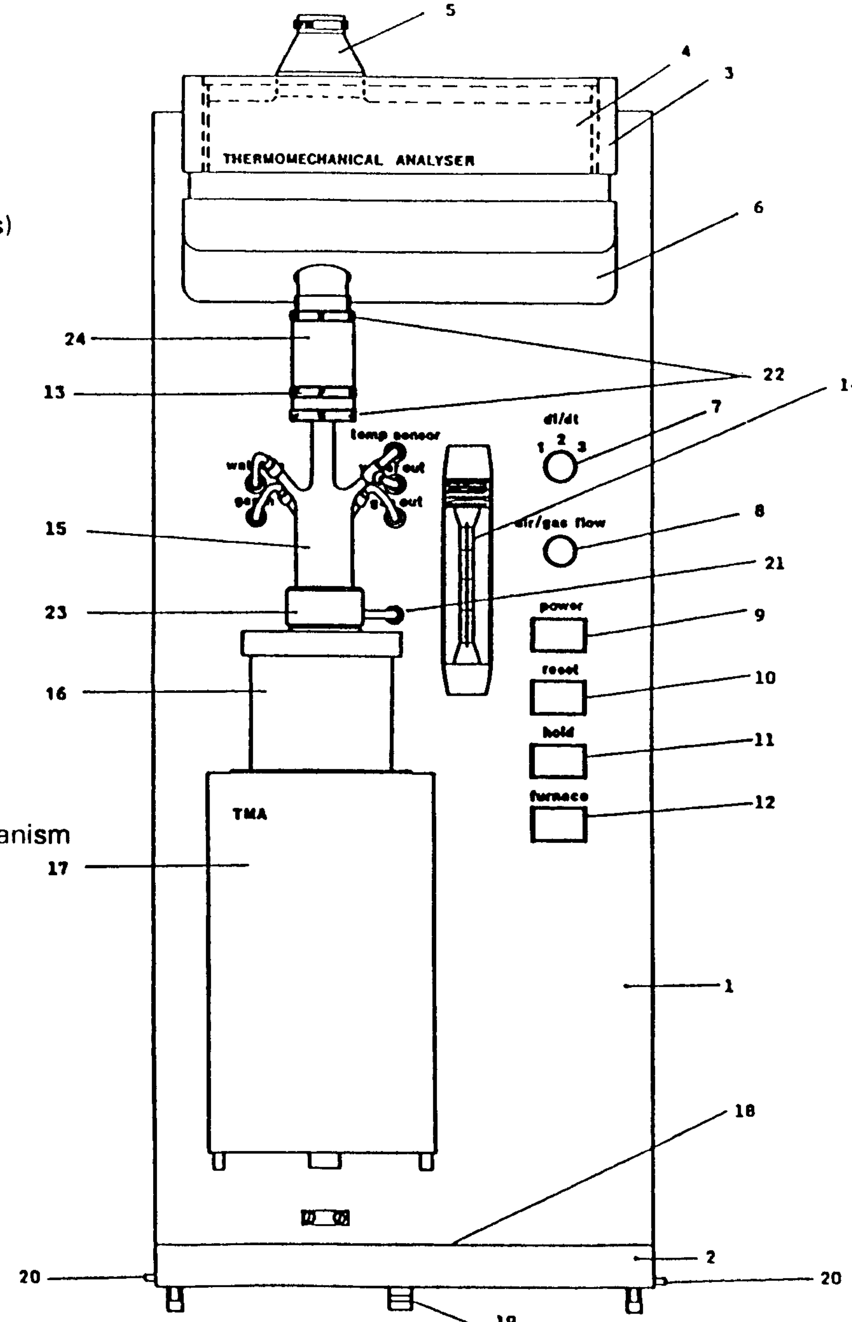

Figure 1. Diagram of Front Main Module

Item	Description
1	Quartz water-cooled jacket
2	Quartz tube
3	Transducer housing
4	Locking screw
5	Threaded portion of screw height adjuster
6	Microswitch connection for motorized furnace mechanism
7	Connection for chromel-alumel thermocouple
8	Chromel-alumel thermocouple
9	Gas and water connections

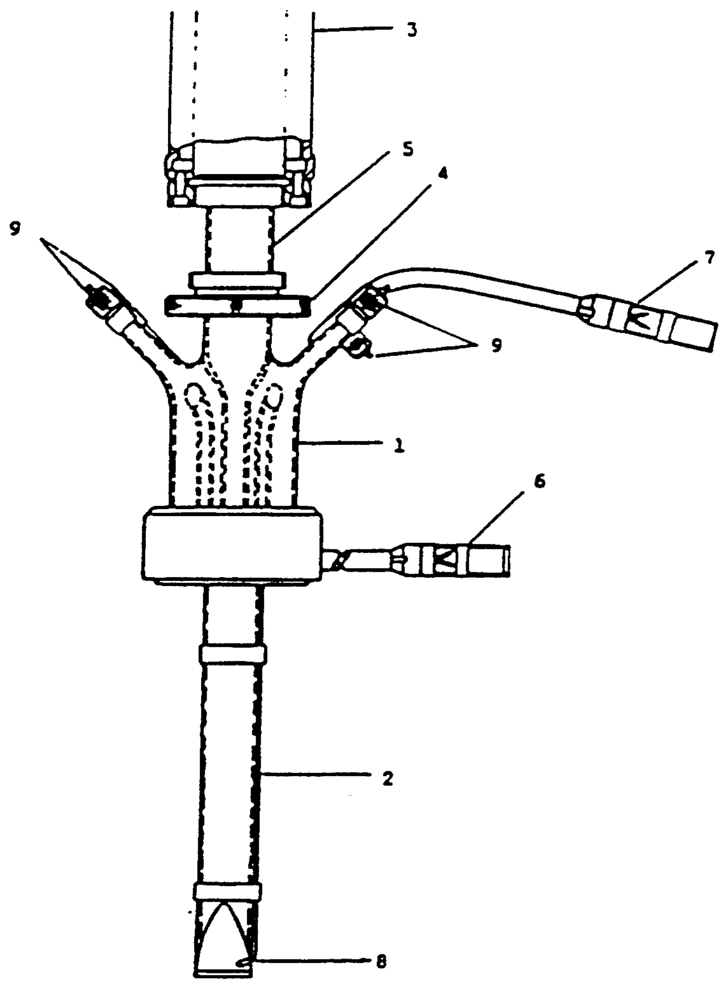

Figure 2. Diagram of Probe Assembly

Item	Description
1	Sample thermocouple
2	Sample
3	Probe
4	Quartz tube
5	Base of quartz tube

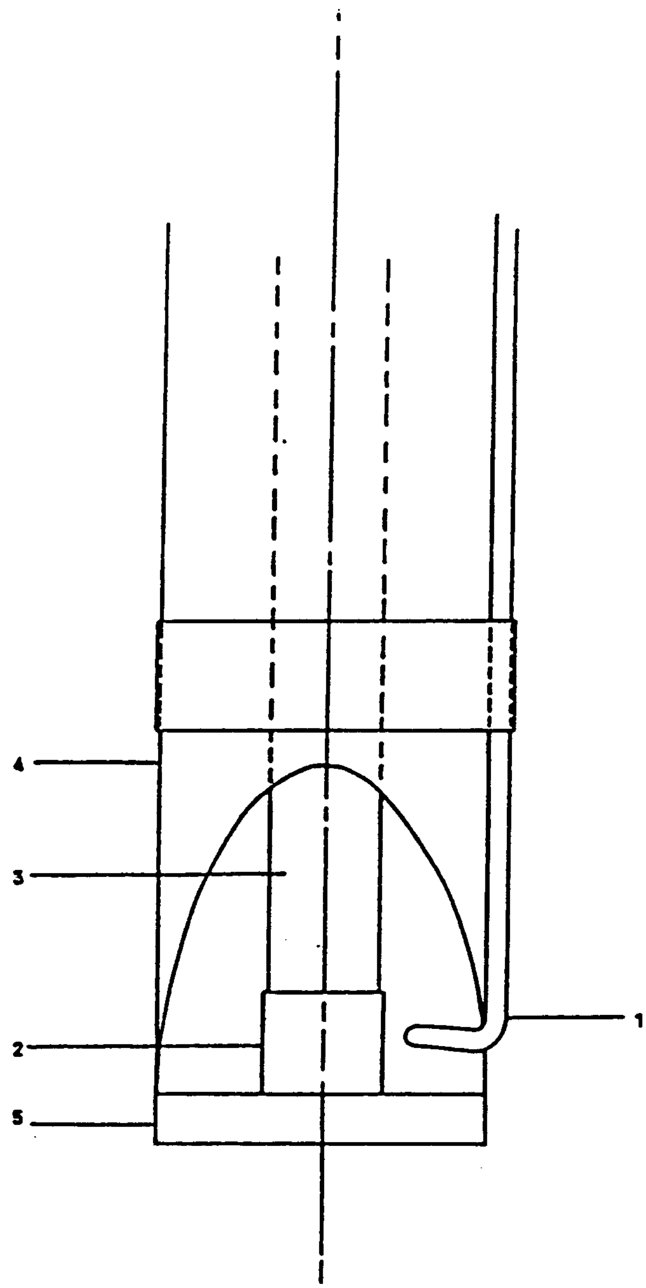

Figure 3. Bottom of Probe Assembly Showing Position of Sample

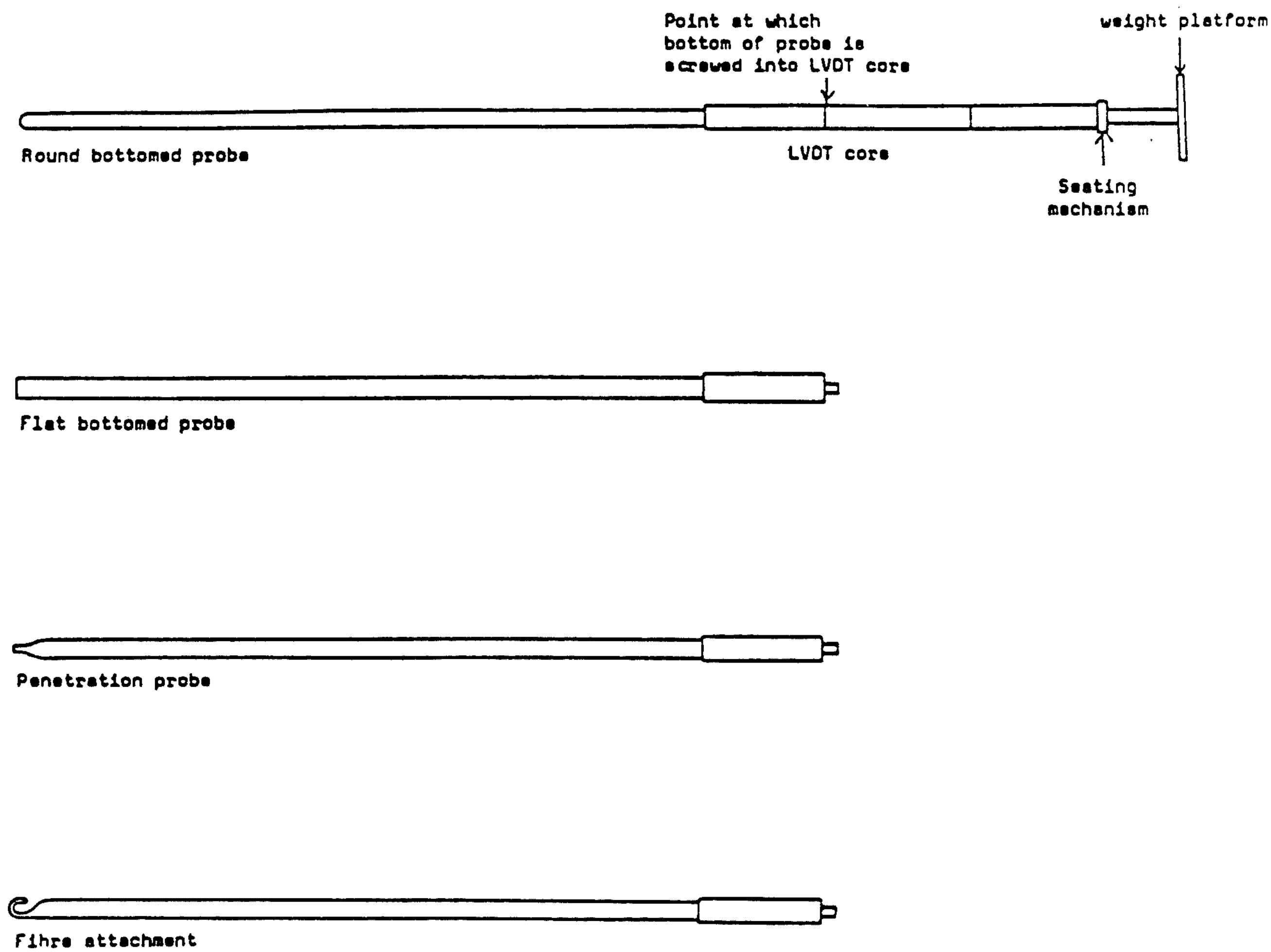

Figure 4. Diagram of Probe Unit Showing Probe Options and Fiber Attachment

Item	Description
1	Microswitch for motorized furnace mechanism
2	Quartz tube of probe assembly
3	Furnace element support tube
4	Water-cooled jacket

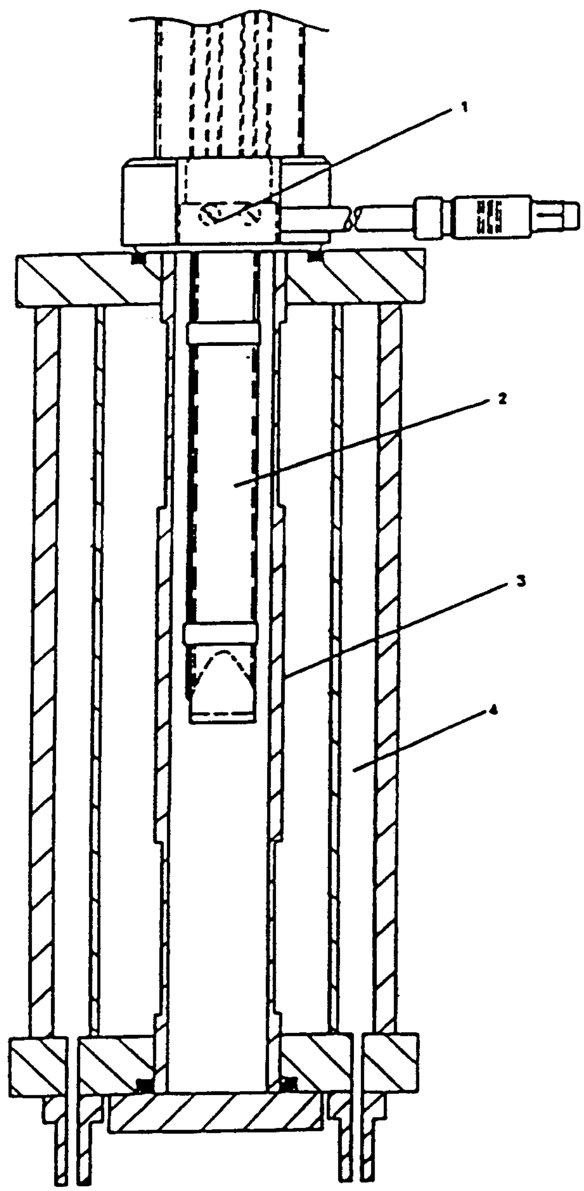

Figure 5. Sectional Drawing of TMA Furnace Assembly and Probe Assembly

3.3 Criteria and Ranking of Indentation Tests.

The following criteria were determined to be important for the selection and ranking of applicable hardness tests.

- Soft-hard range of the material
- Sample thickness (micro-macro tests)
- Equipment/instrumentation: Three levels of identified equipment were handheld, system, and bench.
- Contaminant/decontaminant: The preliminary choice of test and equipment level for contaminants and decontaminants is as follows:
  Contaminants: TMA, handheld, micro
  Decontaminants: TMA, system, bench
- Historical Database Contents: The frequency of various hardness scales in the Chemical Defense Materials Database (CDMD)

Based on these criteria, the following ranking for indentation hardness tests for NBCCS test methodology development was generated and is shown in Table 2. The ranking is listed in column 1, the indentation hardness test in column 2, the test type in column 3, and the equipment level in column 4.

The TMA indentation test is the primary choice for the indentation tests because of the capability to generate temperature-dependent data on micro-sized samples with contaminants and decontaminants. Standardized tests that can be adapted are the ASTM E831 (Linear Thermal Expansion by TMA); ASTM D(TMA 1001a) Tg by TMA; and ASTM E1363 Temperature Calibration of TMA.

Apart from the TMA indentation test, methods that give IRHD values are the primary choice for evaluation. These methods are the ASTM D1415 and its companions ISO 1818, ISO 48, and ISO 1400. These three ISO test methods cover the entire rubber hardness range from low to high, respectively.

The Shore scale is the next candidate for indentation hardness measurements. The ASTM D2240 and ISO 868 for Shore indentation hardness each contain recommendations that specify IRHD as the preferred method for measurement of hardness when used for specification purposes. This further supports the preference of IRHD over Shore hardness.

The next candidates for measurement of indentation hardness for plastics are Ball Indentation (ISO 2039-1) and Rockwell (ASTM D785 and ISO 2039-2). For specification purposes, ASTM D785 is also preferred for plastics and is recommended by ASTM D2240 for hard materials.

Table 2. Ranking of Indentation Hardness Tests for Study of the Effect of Chemical Exposure

Rank	Indentation Hardness Test	Test Type	Equipment Level
1.	TMA Indentation	Micro	System
2a.	IRHD	Micro	Handheld
2b.	IRHD	Micro	System
3.	Shore A, 30-70	Macro	Handheld
4a.	Shore A, 10-35	Macro	Handheld
4b.	Shore A, 10-35	Macro	System
5.	Shore D	Micro	-
6.	ISO Ball Indentation	Macro	-
7.	Rockwell	Macro	-

3.4 Rationale for Indentation Test Ranking.

The selection of hardness methods for rubber over harder materials, such as plastics, was based on the judgement that softer materials would be more prone to degradation in critical performance, such as resiliency of seal for gaskets, o-rings and sealants, etc.

Micro tests are preferred over macro tests. Micro tests require smaller (thinner) specimens and reduce the possibility of errors due to the stacking. Macro tests also require larger quantities of contaminants that could pose safety and disposal difficulties.

Of the equipment/instrumentation levels identified, handheld is preferred for reasons of convenience and cost. The ability to readily move the hardness tester is deemed to be most desirable. In lieu of a handheld tester, a system level tester is the next choice. There is at least one system that meets ASTM D1415 and ISO's 1818, 48 and 1400 for IRHD, and ASTM D2240 and ISO 868 for Shore hardness.

The preliminary match of tests and equipment level suggest the use of a thermomechanical analyzer (TMA) for contaminants and decontaminants. The TMA uses smaller samples, thus requiring less liquid. The TMA can also provide expansion/penetration data over the DoD range of ca. -40 to +160 °F. The approach will be to correlate one or more of the hardness scales to TMA expansion and penetration to provide temperature dependent hardness data for materials before/after exposure to liquid contaminants.

A CBIAC database search demonstrates that of all the tested properties, Shore A/D hardness occurs ca. 10% of the time (one of the four most frequent properties). Correlation and conversion of these values to a less arbitrary scale, such as may be developed by TMA, would be worthwhile.

4. CONCLUSIONS AND RECOMMENDATIONS

Indentation test methodology was reviewed and evaluated with respect to characterizing chemical effects on materials for the Nuclear, Biological, and Chemical Contamination Survivability Hardness criterion in a materials database. Indentation test measurements by thermomechanical analysis (TMA) were concluded to be the most valuable.

For conventional indentation hardness test methods and instruments, it was concluded that the following could be recommended for studies of chemical effects on materials:

- IRHD scale, micro test, softer scale levels versus the harder scales
- Shore A for correlation to prevailing test datasets.